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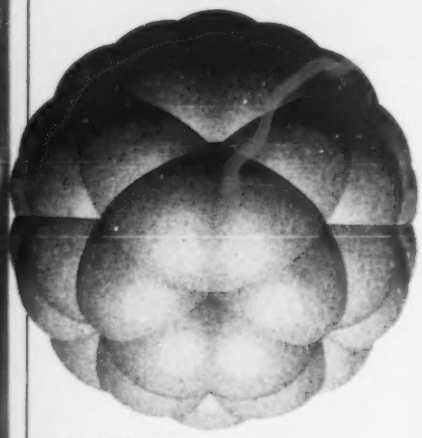

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The International Magazine of Research and Development

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ABOUT THE COVER Researchers are finding new uses for PCR nearly every day. For more on this, see article on page 30. *Photo: Perkin-Elmer Cetus Instruments, Norwalk, CT.*

R&D FOCUS

Uses for PCR Are Multiplying In Gene-Related Research 30
The polymerase chain reaction is proving to be a powerful tool for all researchers who need to amplify sections of DNA.

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Ballistic electron emission microscopy lets you peer into the electronic structure of semiconductor devices.

R&D BUSINESS

U.S. Technology Open to Foreign Investors

Foreign investors who bought into U.S. high-tech industries in the past several years have had little or no opposition, despite creation of a special Congressional Committee on Foreign Investment in the U.S. (CFIUS), says Linda Spencer, a consultant for the Washington, DC-based Economic Strategy Institute.

CFIUS was created in response to the Exon-Florio amendment to the Defense Production Act, passed in August 1988, which empowered the president to block an investment that might harm U.S. national security.

Only one of the 540 investments reported to CFIUS in the past two and a half years received a negative recommendation from the committee. Only 12 cases were formally investigated. The one negative recommendation involved blocking the acquisition of a U.S. aerospace manufacturer by a Chinese company immediately after the Tiananmen Square uprising.

In some investments, foreign firms bought technology vital to U.S. defense interests. In 1990, for example, Japan's Komatsu Electronic Metals Co. acquired Union Carbide Chemical & Plastic Co., the last remaining U.S. supplier of polysilicon. CFIUS chose not to investigate this purchase.

In three of the CFIUS investigated cases, the investments were restructured to ensure that strategic technology was not transferred out of the U.S.

According to a U.S. Government Accounting Office study, CFIUS initially narrowed its scope by limiting its investigations to only those that have direct military impact. In 1990 CFIUS began investigating only those investments where foreign firms became majority shareholders.

For a copy of *Foreign Investment in the U.S.: Unencumbered Access*, 202/728-0993. —*Tim Studt*

U.S. R&D Forecast to Beat Inflation in 1992

The 10 biggest U.S. industrial R&D spenders are forecast to increase their research spending by an average of 7.1% in 1992 over 1991 levels, according to a recent study by Schonfeld & Associates, an Evanston, IL-based management consulting firm.

This compares to only 4% average R&D increases for the 10 biggest Japanese spenders listed on the New York Stock Exchange, which includes NEC, Hitachi, Matsushita, NTT, Toshiba, Sony, and Honda. The top 10 "other foreign" R&D spenders, which includes Philips, Fiat, Imperial Chemical, Northern Telecom, Glaxo, and Smith Kline Beecham are forecast to increase their average research spending by 8.8%.

Many segments of the computer industry will increase their research spending between 6.5% and 15%, according to the study. However, most increases will be in software development where total R&D spending will exceed $2 billion.

R&D Ratios & Budgets ($295) lists spending forecasts for more than 3,000 companies and 300 industries. For a copy: 708/948-8080. □

U.S. Firms Renew R&D Commitments

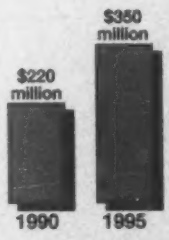
	Top U.S. R&D spenders	1991* (In billions)	1992** (In billions)	Percent change
1	General Motors	$5.76	$6.09	5.6%
2	IBM	5.44	5.79	6.4
3	Ford Motor	3.89	4.22	8.6
4	AT&T	2.41	2.40	-0.4
5	Digital Equipment	1.96	2.16	10.1
6	Hewlett-Packard	1.58	1.74	10.4
7	General Electric	1.58	1.68	6.6
8	DuPont	1.51	1.58	4.6
9	Eastman Kodak	1.46	1.57	7.6
10	Dow Chemical	1.27	1.42	11.5

*Estimated **Forecast

Source: Schonfeld & Associates

General Motors is forecast to be the first company to spend more than $6 billion in R&D. Dow Chemical is the only newcomer to this top 10 list; all others were in 1990's list.

International Sales Drive FTIR Growth



European and Pacific Rim demand for Fourier-transform infrared (FTIR) instruments will drive 9.6% annual growth to $350 million in 1995, according to Strategic Directions Intl., a Los Angeles-based management consulting firm.

This growth reflects FTIR replacement of dispersive-IR techniques due to FTIR's greater analytical capabilities and lower price. FTIR is used in R&D and forensics applications.

FDA Is Biggest Barrier to Biotech Firms' Success

The U.S. Food and Drug Administration was ranked number one of 22 possible barriers to the success of U.S. biotech firms, according to a recent survey by the North Carolina Biotechnology Center, Research Triangle Park.

The survey of 166 U.S. biotech firms included external factors, such as financing sources and government regulations, and internal factors, such as management expertise and the availability of personnel. The factors that received the highest ranking were:

U.S. Food and Drug Administration
U.S. patent decisions
Management expertise
Federal regulations
Availability of technical personnel
Foreign patent decisions
R&D technical expertise
Availability of marketing personnel

The lowest-ranked barriers, or those factors that were not considered to limit the success of a biotech firm, included the availability of materials, the U.S. Dept. of Agriculture, local government regulations, seed funding, government research funds, and bioprocessing expertise. R&D

Researchers See Bright Prospects for New STEM

The Cavendish Laboratory, Cambridge Univ., England, has a new approach to scanning transmission electron microscopy (STEM) that could lead to major improvements in our understanding of microstructures.

"The idea is to record diffraction patterns and images simultaneously, so that a computer program can combine the four-dimensional information to give you higher resolution," says L.M. Brown of the lab's Microstructural Physics Research Group.

The researchers believe they can improve the spatial resolution of STEM from 0.5 nm to better than 0.1 nm.

U.S. Must Revamp R&D Funding, OTA Says

The government of the United States, facing increased global competition and domestic financial pressures, must re-evaluate its priorities for R&D funding, says the Office of Technology Assessment (OTA), a congressional agency.

In the report "Federally Funded Research: Decisions for a Decade," the OTA offers recommendations for solving problems in Federal funding for scientific research.

The report urges Congress to demand that the Bush Administration set priorities within and across agencies for science megaprojects. It says that criteria are unclear for megaprojects such as the superconducting supercollider, the human genome project, and the manned space station (which the OTA says has "little justification on scientific grounds"). The OTA also says there is no way to evaluate the total Federal government research portfolio across agencies in terms of national objectives.

Furthermore, there should be greater cost-accountability, especially regarding allowable indirect costs (overhead), and better estimates of future expenditures, particularly for megaprojects. One reason for this problem is rising research costs, which are outpacing available Federal funds, the OTA says.

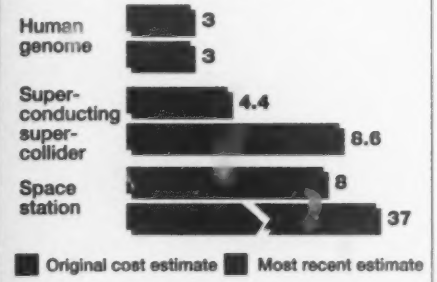
Agencies also should refine internal data collection and analysis to improve research decision-making, according to the report.

The OTA concludes that implementing these and other changes can help government officials make the "tough choices, even beyond merit and constricted budgets, in guiding the research system."

For a copy: Stock #052-003-01241-3 ($12); 202/783-3238. —*Ted Agres*

U.S. Science Megaprojects Cost Megabucks

(In billions of dollars)



Overall costs for certain Federally funded science projects are enormous, says the OTA. Over time, cost estimates can even quadruple for some megaprojects, such as the manned space station.

Scientists Best Fish In Making Antifreeze

Winter flounders don't freeze in temperatures as low as 27 F because their bodies produce a natural antifreeze peptide (AFP) that is about 500 times more effective in preventing ice formation than an equivalent concentration of sugar or salt. So why doesn't someone create a synthetic version?

Well, someone has, and the payoff could be new ways to prevent crops from freezing, noncorrosive substitutes for road salt, and coatings to prevent ice formation on aircraft wings in bad weather. Another use may be as a cryoprotective package for enzymes.

Researchers at Virginia Polytechnic Institute, Blacksburg, have synthesized AFPs that mimic the structure of the antifreeze compound the fish produce but are larger and should work at even colder levels, says Thomas Caceci of the school's College of Veterinary Medicine. The group is the first to design and synthesize an AFP.

Artificial antifreeze peptide research by Thomas Caceci's group may result in alternatives to road salt and coatings to prevent ice formation on aircraft.

"Activity seems to depend on molecular size and the number of places the AFP can contact ice," says Caceci, who heads the research team. "We know they can protect at lower than 27 F, but we really don't know yet what the lowest temperature attainable is."

The researchers postulate that the natural AFP suppresses ice forming inside the fish by binding to ice crystal

Breaking Down the Barriers for Ceramic Matrix Composites

Advances in toughening systems for ceramic composites are enhancing use in industrial high-temperature, wear, and corrosive applications.

By Tim Studt, Senior Editor, R&D Magazine

R&D

Ceramics are almost the perfect structural materials. They're light, strong, hard, chemically inert, and resistant to abrasion. They can withstand extremely high temperatures. Their raw materials are abundant. They really only have two weaknesses: they're brittle and hard to work with.

Researchers are working on these weaknesses with surprising success. The solution has been to make ceramic matrix composites (CMCs).

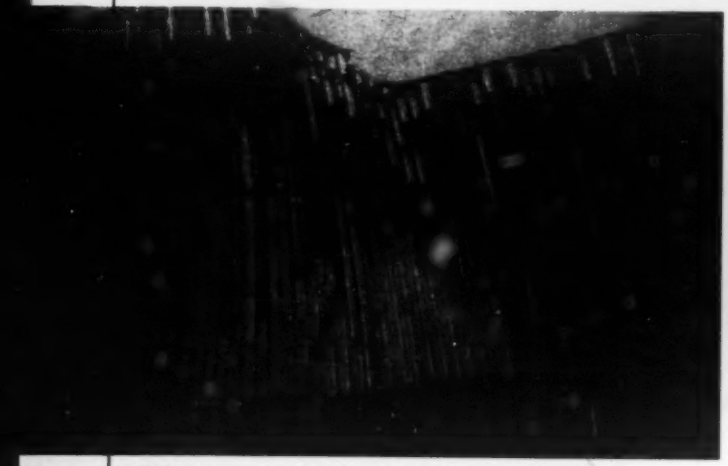
The great potential of CMCs has only begun to be recognized as production scale-up processes prove themselves out and as the first parts find their way into industrial applications—cutting tools, automotive and aerospace engine parts, heat exchanger tubes, and heavy-duty components in process equipment.

Researchers now find that CMC products not only can improve upon and replace existing ceramic parts, but they also can replace metal and even metal matrix composite parts.

Improving the fracture toughness of ceramics started with rudimentary attempts at adding particulates, platelets, and ceramic whiskers. Current technologies include improved versions of these reinforcements and add continuous fibers, matrices created by vapor infiltration and directed metal infiltration, and various types of laminated structures.

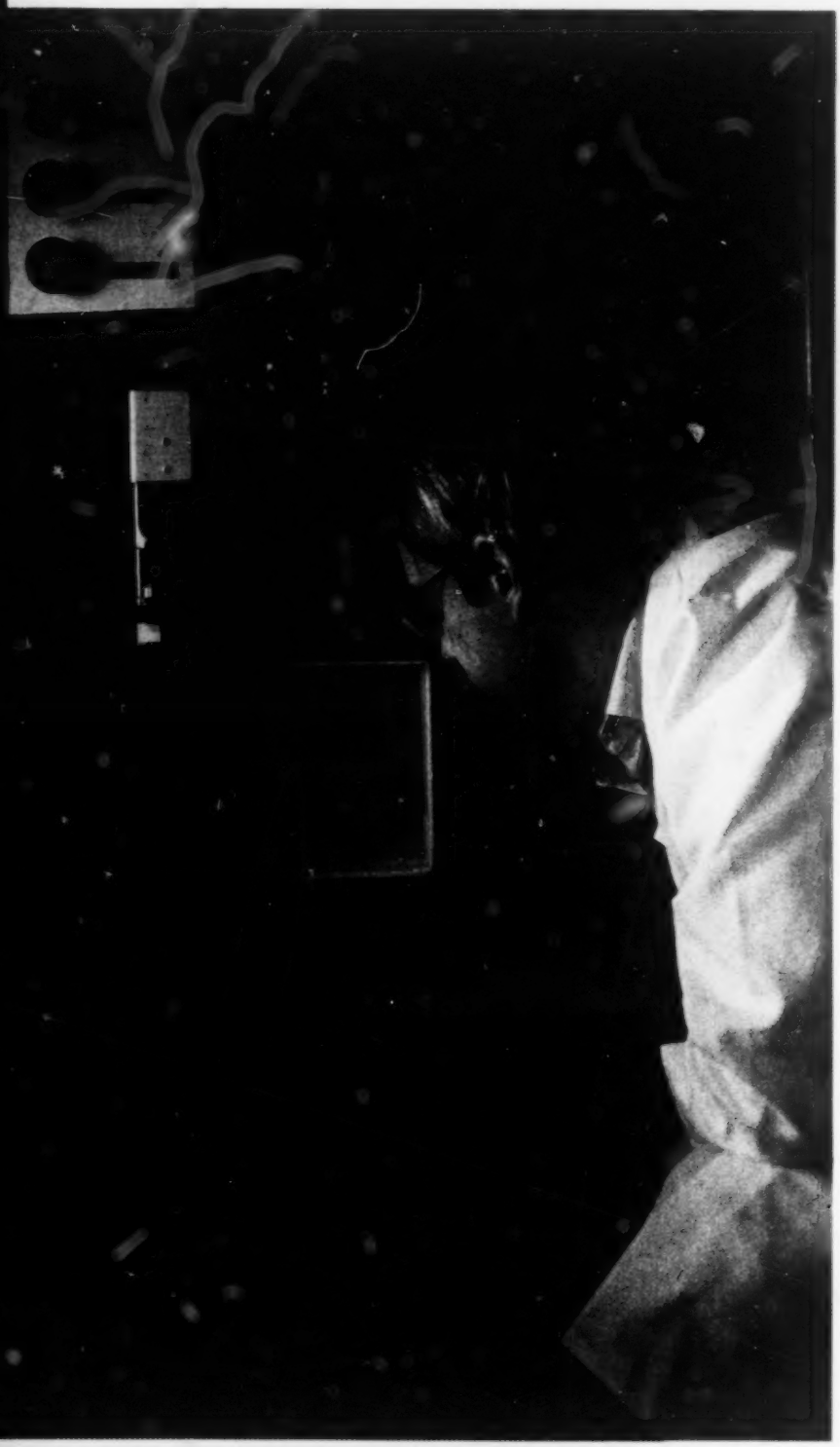
"Everyone knows you can improve the performance of a monolithic ceramic by incorporating whiskers into the matrix," says Paul Becher, a researcher in the Metals and Ceramics Division at Oak Ridge (TN) National Laboratory (ORNL). "However, if you have a whisker-like grain structure you can do the same thing, which is taking a little bit of what we learned in the composite area and extending it to monolithic materials."

Whisker-reinforcing technology has improved to the point where it cur-



***SEM picture of a fractured** SiC-fiber-reinforced Si_3N_4 reveals poor strength in the through-thickness direction.*

***Higher magnification** of the fracture at left reveals a planar matrix fracture and failure of the fiber coating.*



Paul Becher, ceramic researcher at Oak Ridge (TN) National Laboratory, tests the high-temperature strength of a whisker-reinforced ceramic matrix composite.

rently is the most economical fabrication method for improving the fracture resistance of a ceramic product. Japanese firms already use whisker-reinforced SiN materials to make automotive turbocharger rotors.

"The whisker raw material cost is still high because you're not making a million parts a year," says Becher. "But you can use fairly standard fabrication technology, such as that used for standard powder processing of monolithic ceramics."

Research in SiC whisker and platelet reinforcements focuses on their high-temperature behavior and production scale-up capabilities. There also is research on reducing the size of platelet reinforcements from 50- to 100-μm across to 10 μm or less across. Large platelets often act more as a flaw than as a reinforcement.

Silicon carbide also is used in most continuous-fiber CMC products. The advantages of SiC are its chemical and thermal inertness to many matrix materials, its oxidation resistance, and its strength at high temperatures.

Continuous SiC fiber-reinforced CMCs have up to five times the fracture toughness of monolithic products. They also have a more gradual failure mode than the catastrophic brittle failure typical of monolithic and whisker-reinforced products.

There are two types of SiC fibers available. Small diameter (10 to 15 μm) fibers, such as Nippon Carbon's Nicalon and 3M's Nextel, are relatively inexpensive and can be woven. Large diameter (140 μm) fibers, such as Textron's SCS6, are stiff and hard to work with, but have better strength and high-temperature properties.

Nicalon-type fibers are used more for industrial CMC applications, while SCS6 fibers are used for advanced aerospace applications.

Research in new fibers is directed at further reductions in the oxidation of these SiC fibers. Unless protected or treated, SiC oxidizes steadily when heated to more than 1000 C.

The high temperatures and mechanical stresses that the fibers experience during the fabrication of a continuous fiber-reinforced CMC often limits the overall composite's performance. Researchers are looking for ways to reduce these stresses and temperatures to limit the degradation of the fragile SiC fibers.

"We blended an additive with the

Continuous-fiber-reinforced ceramics have up to five times the fracture toughness of monolithic ceramic materials.

silicon nitride matrix of a hot-pressed SCS6-reinforced part to match the thermal expansion mismatch between matrix and fiber," says Jeff Bright, a research engineer at Ceramatec, Salt Lake City. "With this process, we were able to lower the press temperature 200 C, cut the process pressure by more than half, and still fabricate a composite that can withstand a 300 MPa load at 1400 C."

Narottam Bansal, a materials research engineer at NASA's Lewis Research Center, Cleveland, also works with additives to stabilize a SCS6-reinforced celsian glass CMC. "With this process, we can fabricate a hot-pressed CMC part with an ultimate strength of 500 MPa at room temperature in air."

However, even these fabrication techniques still impart significant stress to the fibers. More research is being performed on the development of lower stress CMC processes.

Chemical vapor infiltration (CVI) is one of the most highly commercialized of these low stress processes. In the CVI process, the CMC matrix is created by reacting two or more vapor species in a heated furnace. The matrix infiltrates and forms on the surfaces of a reinforcing fiber preform.

DuPont is the U.S. licensee of the French Société Européenne de Propulsion (SEP) CVI process. DuPont fabricates both continuous-SiC and continuous-carbon fiber-reinforced SiC CMC products in their CVI process.

Other ceramatists are developing their own CVI processes. Researchers at ORNL have developed a forced-flow process that reduces infiltration time and creates thicker parts.

Researchers at NASA's Lewis Research Center are developing a CVI process that uses low-pressure mixtures of $SiCl_4$ vapor and H_2 gas to deposit silicon throughout a microporous carbon preform, resulting in a thick SiC product.

But DuPont already is making production parts with its process.

"We licensed CVI to solve the problems of scale-up from laboratory-type furnaces to production furnaces," says Warren Hewett, CMC research manager at DuPont, Newark, DE. "We think the design of the furnace is the key to achievement of uniform and reproducible parts."

Most of DuPont's products are for high-speed aircraft, liquid propulsion rockets, and small expendable turbine engines.

"We also are targeting industrial markets, such as those for heat exchangers, where we can provide parts that operate at up to 1100 C for hundreds of hours," says Hewett. "We've already made a 0.6-m × 0.6-m aerospace part and are planning a furnace that can accommodate parts 1.5-m-dia × 2-m high."

Even in CVI, the reinforcing fiber limits how long the CMC survives at an elevated temperature.

"To improve our current SiC/SiC and C/SiC systems, we need to see improved SiC and carbon fibers," says Hewett. Both Nippon Carbon (Nicalon SiC fiber) and Ube (Tyranno Si/Ti/C/O fiber) are developing new fibers with reduced oxygen contents for better high-temperature capabilities.

DuPont also is involved

Forecast for U.S. Ceramics Markets

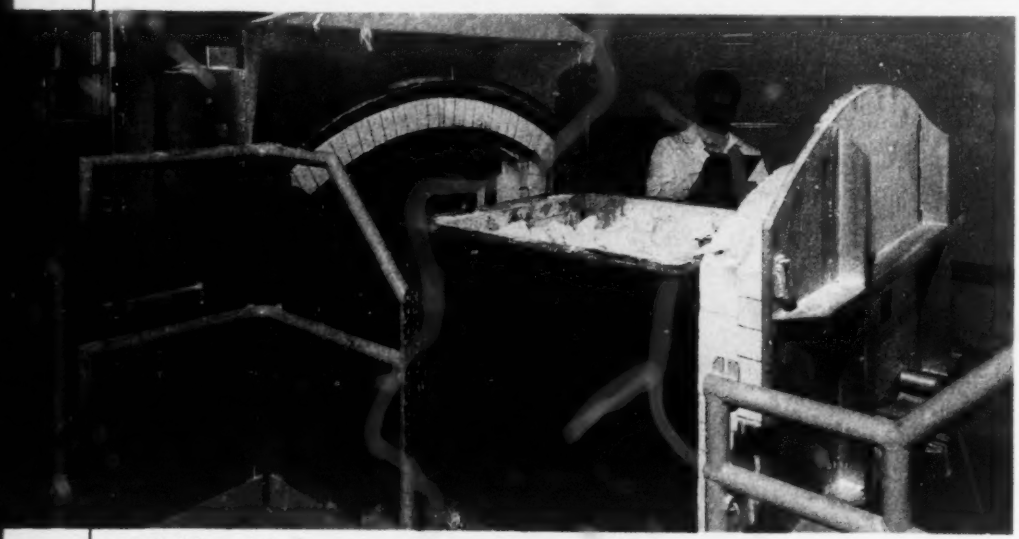
(Millions of current dollars)

	1990	1995	2000
Electronics applications	3,740	6,565	11,360
Structural applications			
Aerospace and defense-related	80 (28)*	200	445
Automotive	81	310	820
Bioceramics	15	34	60
Cutting tools	92 (19)*	246	500
Heat exchangers	15	50	100
Wear parts and other industrial	150 (32)*	320	720
Subtotal (structural)	433 (79)*	1,160	2,645
Grand total	4,173	7,725	14,005

* Est. CMC portion

Source: U.S. Bureau of Mines

***Experts predict that improvements** in the high-temperature performance of CMCs will increase their current $79 million or 18% share of the structural ceramic market.*



***A technician at Carborundum,** Amherst, NY, reviews in-process step in the manufacture of boron nitride. Blending zirconia with BN substantially increases its toughness.*

Chemical vapor infiltration is the most highly commercialized of the low-stress ceramic matrix composite processes.

in a joint venture with Lanxide Corp., Newark, DE, to develop directed metal oxidation (DMO) technology for fabricating fiber-reinforced CMCs.

In the DMO process, the matrix is grown by reacting a molten metal vapor with an oxidant. The metal is progressively drawn through its own oxidation product, resulting in a ceramic product.

"We use aluminum to make alumina or aluminum nitride, depending upon the reaction that takes place, or we use zirconium to make zirconium nitride or zirconium diboride," says Michael Hollins, a vice president at Lanxide. "Most ceramics tend to be the result of reactions between metals and some kind of reactant."

"We've worked with aluminum, zirconium, titanium, silver, hafnium, and tin," he says. "If there is a useful reaction product of a metal, there is a good probability our technology can be applied to a metal precursor to obtain a ceramic product."

The DMO process has been used to make 2-m-long heat exchanger tubes, rocket motor components, and wear- and corrosion-resistant components for the mining and chemical process industries.

Another low-stress process is being developed by researchers at NASA's Lewis Research Center. Here, a liquid Mo-Si alloy infiltration process, developed by Yet-Ming Chiang at Massachusetts Institute of Technology, Cambridge, is used to create an SiC-fiber-reinforced SiC matrix by infiltrating silicon into carbon preforms.

"The Mo-Si alloy converts the carbon to silicon carbide and the molybdenum shows up as molydisilicide in the pores of the preform," says Donald Behrendt, a research scientist at Lewis Research Center. "The Mo-Si eliminates any free silicon that might remain in the final material."

$MoSi_2$ also is the most oxidation-resistant material of all the silicides. Behrendt coats the SCS6 fibers in this CMC with BN to protect the fiber from the molten silicon.

NASA Lewis researchers also are working on a similar reaction process for forming a silicon nitride CMC matrix. In this process, the existing carbide coating on the SCS6 fiber can be used to protect the fiber.

continued

Tougher Testing for Toughened Ceramics

Some of the characteristics that make ceramics so useful—hardness, stiffness, and thermal stability—also complicate the testing of these materials. To prevent a stray bending load from distorting a test result, for example, researchers can take more than a week aligning a ceramic specimen in a conventional tensile test fixture. Additionally, most tests need to be run at high temperatures.

Creating a more fracture-resistant ceramic matrix composite (CMC) further complicates this testing. Fiber-reinforced CMCs, for example, don't follow predictable failure rules. Different failure modes often occur within the normal operating temperature range of a CMC.

Chemical changes in the composite at elevated temperatures, such as oxidation of SiC reinforcing fibers, add even more variables.

Bend tests have been the primary method for measuring the strength characteristics of ceramic materials because these tests are simple and cost effective. However, bend tests don't supply all the strength data you need to design a new product.

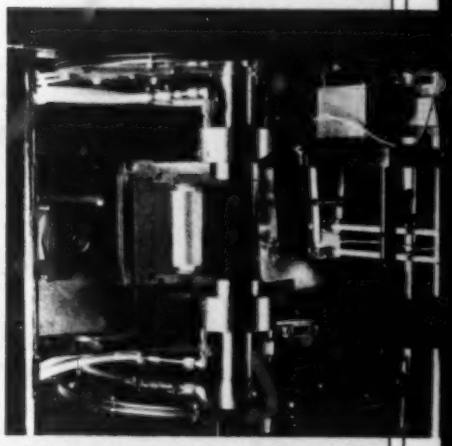
Self-aligning grip systems recently have been developed that eliminate bending loads in the more comprehensive tensile tests. These grips allow you to obtain more information on CMCs quickly, economically, and at temperature.

"We tensile test CMCs up to 1600 C and we have ideas that will allow us to test at 1800 C," says David Jablonski, manager of the research lab at Instron Corp., Canton, MA. "We have requests to test to 2300 C, but we have not done that yet."

The hardness and wear-resistance at temperature of CMC materials force yet more new test fixtures to be developed. Christopher Della-Corte and Serene Farmer, researchers at NASA Lewis Research Center, Cleveland, developed a special apparatus that measures the friction and wear behavior of a SiC-whisker-reinforced alumina at 1200 C. This information helps determine if CMCs can be used to make automotive engine cylinder liners.

Measuring the fracture toughness of CMCs is difficult and often confusing. No standardized method has yet been developed. The Vickers indenter method is the most widely used method because it's fast and inexpensive. It's also more acceptable than putting a notch in a notch-sensitive material.

In all these cases, electron microscopy of the failed CMC section is used to correlate the measured physical values with the actual reason for the ultimate failure. In monolithic ceramics you look for defects as the primary cause of failure. In CMC materials, you also look for problems in the reinforcement, the interfaces, and the reinforcement coatings.



High-temperature test system with capacitive extensometer, short furnace, and special grip system allows tensile test of ceramics without bending loads.

NASA Lewis Research Center technician loads a ceramic tensile test specimen in a sealed environmental test chamber.

'We licensed ceramic technology to solve the problems of scale up from lab to production,' says DuPont's Warren Hewett.

Another ceramic-toughening concept being developed is to laminate different ceramic materials. Researchers at Ceramatec created a three-layered composite consisting of 200-μm-thick monoclinic Al_2O_3-ZrO_2 outer layers encasing a tetragonal Al_2O_3-ZrO_2 core.

This process relies on the retention of surface compressive stresses in the skins to toughen the product. Under a load of 1,000 N, test samples had seven times the compressive strength of a comparable monolithic product.

"We're making three-layered cam followers for automotive applications," says Raymond Cutler, a scientist at Ceramatec. "However, manufacturers want this system to be cheaper than metal parts."

Researchers at the Center for Advanced Materials, Pennsylvania State Univ., University Park, also have fabricated laminated ceramic composites to improve fracture toughness. Maurice Amateau and Gary Messing found that a laminated SiC-whisker-reinforced alumina composite with high residual compressive stresses in the skin had more than twice the compressive strength of a comparable monolithic product.

Still another toughening method is being looked at by Waltraud Kriven, professor of materials science at Univ. of Illinois, Champaign. In this case, a material that undergoes phase transformations under stress, such as dicalcium silicate or a lanthanide sesquioxide, is added to the ceramic as a toughening agent.

"We allow the ceramic to crack in such a way that the crack uses up some of its energy in forming a particular type of basic crystal structure," says Kriven.

Zirconia parts with this additive had three times the toughness of nonreinforced zirconia parts. "The composite structure helps the zirconia keep its shape, confining the transforming explosions to pushing on the crack and closing it," she says.

Zirconia by itself also can be used as a toughening agent for other ceramic materials. Researchers at Carborundum, Niagara Falls, NY, added zirconia and SiC to toughen normally brittle boron nitride. The resulting glassy ceramic is stronger and more corrosion resistant than boron nitride, while retaining BN's high temperature and electrical performance.

Further refinements to these toughening processes and material developments should continue to drive down the overall cost of CMCs and create even more applications. **R&D**

O. Omatete (left) and Mark Janney, researchers at Oak Ridge (TN) National Laboratory, demonstrate a gelcasting process they use to make ceramic rotors for gas turbines.

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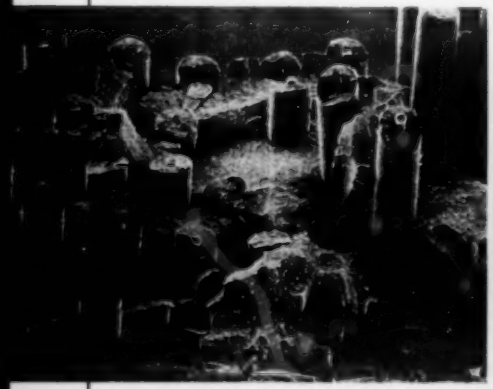
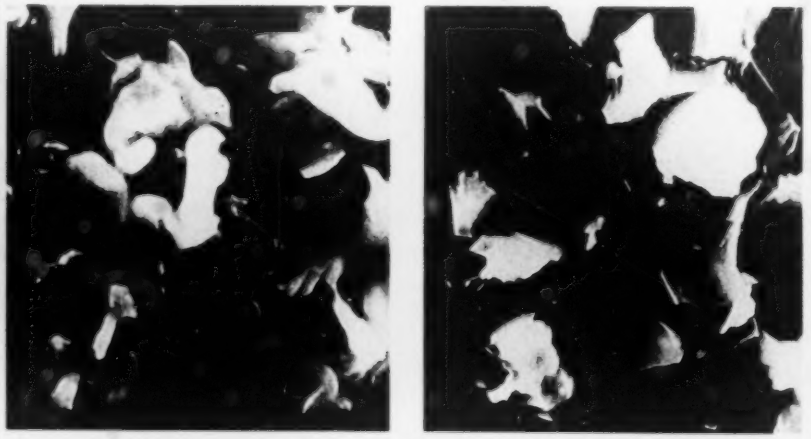


***Fracture surface** of a hot-pressed SiC-fiber-reinforced celsian glass composite reveals the fiber pull out that toughens the brittle glass matrix.*



***Fracture surface of porous carbon** (left) is compared with a similar fracture surface after being infiltrated with Mo-Si alloy (right) creating a SiC matrix with excess silicon.*

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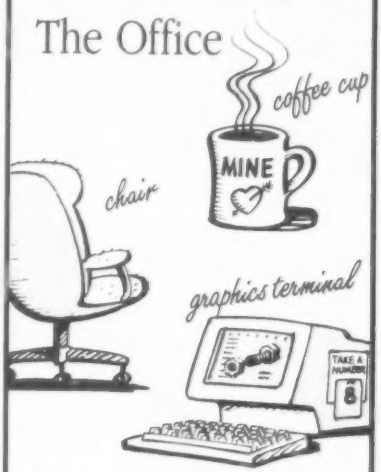
ABP BPA

Three Things You Shouldn't Have To Share At The Office



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